

DECLARATION UNDER 37 CFR § 1.132

1. The following experimental data is being submitted in response of an office action issued on March 31, 2010

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2. This experiment is conducted for proving that it is necessary to satisfy the ratio of the organic peroxide to triallyl isocyanurate is in the range of 60:40 to 52:48 in the sheet-shaped adhesive containing the peroxy carbonate having a formula I in the specific amount, in order to obtain excellent torque, penetration resistance, adhesion and reduced haze and variation of hazes.

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3. Experiments

[Example I]

Raw materials having the following formulation were processed by calendaring process as shown in Fig. 2 of the present specification to prepare a sheet-shaped transparent adhesive (thickness: 400μm). The raw materials were kneaded at 80°C for 15 minutes, and the calendar rolls had a temperature of 80°C and the processing rate was 5m/min.

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(Formulation for forming sheet-shaped adhesive)

EVA (content of vinyl acetate: 25 wt. %) : 100 weight parts

Crosslinker (t-butylperoxy-2-ethylhexylcarbonate)

: 2.5 weight parts

Triallyl isocyanurate (abbr. TAIC)

: 2.0 weight parts

Silane coupling agent (3-methacryloxypropyl trimethoxy silane)

: 0.5 weight part

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<Preparation of a laminated glass>

Two silicate glass plates having thickness of 5 mm, which was preliminarily washed and dried, were prepared as glass plates. The sheet-shaped adhesive was inserted between the two glass plates, and the glass plates having the adhesive was put into a rubber case to be degassed in a vacuum and then preliminary bonded at 110°C under pressure. The bonded glasses was introduced into an oven and heated at 130°C

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for 30 minutes, and then cooled such that the atmosphere temperature is dropped at rate of 20/min. to prepare a laminate (a laminated glass) of the invention.

[Examples II to V]

5 The procedures of Example I were repeated except for changing the formulation for forming sheet-shaped adhesive to that shown in Table I to prepare a laminate (a laminated glass) of the invention.

[Comparison Examples I to III]

10 The procedures of Example I were repeated except for changing the formulation for forming sheet-shaped adhesive to that shown in Table I to prepare a laminate (a laminated glass) for comparison.

<Evaluation on laminate and sheet-shaped adhesive >

15 (Cure torque)

Bulk materials having 5cm^3 are prepared in the same manner as in Examples I to V and Comparison Examples I to III. The cure torque is determined by heating each of the bulk materials having 5cm^3 to 130°C , and starting measurement of a cure torque and measuring a cure torque after 30 minutes from the starting point in time by using a
20 cure torque meter (IV type; available from JSR Corp.). The cure torque was determined according to JIS K 6300-2-2001.

(Penetration resistance)

Transparent laminates (Examples I to V and Comparison Examples I to III) obtained by inserting the sheet-shaped adhesive between two glass plates are subjected
25 to a penetration test according to JIS R 3205 (1985). Penetration resistance is evaluated as follows:

A: The outside glass plate (impacted plate) of the laminate is passed through but the inside glass plate and the sheet-shaped adhesive are not passed through.

C: The outside glass plate of the laminate is passed through and the inside
30 glass plate and the sheet-shaped adhesive are also passed through.

(Adhesive strength)

Laminates are prepared in the same manner as in Examples I to V and Comparison Examples I to III except for using PET film having thickness of 50mm.

The laminates are cut to prepare test pieces having width of 2.5cm and length of 10cm. The test pieces are subjected to measurement of 180° peeling strength between glass plate and adhesive at tensile rate of 100mm/min. and temperature of $22 \pm 5^{\circ}\text{C}$ by using a tensile tester. Three test pieces per each Example are measured to report the mean value as the adhesive strength. The measurement is carried out carried out according to JIS K 6854 (1994).

(Haze)

Hazes of the laminates prepared in Examples I to V and Comparison Examples I to III are measured according to JIS 7105(1981). In more detail, haze values of three test pieces of each Example are measured by using Color Computer SM-5 (available from Suga Test Instruments Co., Ltd.). The mean value of the three test pieces is reported as haze.

(Haze ratio)

Laminates are prepared in the same manner as in Examples I to V and Comparison Examples I to III except for changing the thickness of the sheet-shaped adhesive to $400\mu\text{m}$ and $1,600\mu\text{m}$. Two hazes of the laminates ($400\mu\text{m}$ and $1,600\mu\text{m}$) of each Example are obtained in the same manner as above, and their percentage is calculated.

The obtained results are shown in Table I.

Table I

	Example					Comparison Example		
	I	II	III	IV	V	I	II	III
Perbutyl E	2.5	3	2.2	3	2.2	1.5	2.5	4
TAIC	2	2	2	2.76	1.47	2	1.5	2
Coupling agent	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Peroxide portion	55.6	60.0	52.4	52.1	59.9	42.9	62.5	66.7
TAIC portion	44.4	40.0	47.6	47.9	40.1	57.1	37.5	33.3
Cure torque	108	110	105	113	100	88	103	119
Penetration resistance	A	A	A	A	A	C	C	C
Adhesive strength	13	12	14	12	14	19	16	16
Haze	0.2	0.2	0.2	0.2	0.2	0.4	0.3	0.4
Haze ratio	230%	230%	202%	210%	280%	306%	325%	317%

Note; Perbutyl E (Peroxide) : t-butylperoxy-2-ethylhexylcarbonate

As apparently indicated from the above results, the sheets obtained in Examples I-V containing the peroxy carbonate having a formula I in the specific amount and satisfying the ratio of the organic peroxide to triallyl isocyanurate in the range of 60:40 to 52:48 showed excellent torque, penetration resistance, adhesion and reduced haze and haze variation. In contrast the sheets obtained in Comparison Examples I-III containing the peroxy carbonate having a formula I and not satisfying the ratio of the organic peroxide to triallyl isocyanurate in the range of 60:40 to 52:48 are inferior in penetration resistance and reduced haze and haze variation to the Examples. Although the Examples are a little inferior in adhesion to the Comparison Examples, the adhesion has no problem from a practical standpoint.

I, Masao Hashimoto, attached to Performance Film Development Department of Bridgestone Corporation, hereby verify that the foregoing data was obtained in an experiment conducted on October 27, 2009. I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief
5 are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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June 29, 2010
Date

Masao Hashimoto
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